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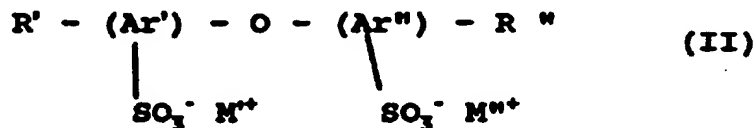
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(54) Title: CLEANING COMPOSITIONS

(57) Abstract

A concentrate for use in diluted form in an aqueous cleaning composition for cleaning soft metals, which concentrate comprises the following components: (a) a source of calcium ions; (b) 2-phosphonobutane-1,2,4-tricarboxylic acid or a salt thereof (PBTC); and (c) (i) a saturated polymer containing carboxylate groups; (ii) a sulphonate compound of the general formula (I): $R - (Ar)_n - SO_3^- M^+$ in which Ar is phenyl or naphthyl, $n=0$ or 1, M^+ is H^+ or a suitable counterion, and R is a C_8 to C_{18} straight chain alkyl group and/or (iii) a sulphonate compound of general formula (II), in which Ar' and Ar'' are the same or different and are phenyl or naphthyl, $M^{'+}$ and $M^{''+}$ are the same or different and are H^+ or a suitable counterion, and R' and R'' are the same or different and are C_6 to C_{16} straight chain or branched chain alkyl.



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CLEANING COMPOSITIONS

The present invention relates to cleaning compositions and more particularly, to soft metal safe alkaline cleaning compositions.

Alkaline cleaning compositions are particularly useful in the removal from various surfaces of tenacious soils, particularly those containing biological material such as proteins and fats. Where the surfaces to be treated by the alkaline cleaning compositions are made of soft metals such as aluminium, the causticity of the alkaline cleaning compositions tends to cause corrosion of the soft metal surfaces. It has been common practice to use sodium silicate as a corrosion inhibitor in these alkaline cleaning compositions. However, the causticity of such "silicated" cleaning compositions must be kept low, otherwise the compositions become unstable and unsafe to apply to the soft metal surfaces. This has limited the use of such silicated compositions to light-duty cleaning only. Additionally, silicated compositions deposit a white silicate film on the metal surfaces thereby posing rinsing difficulties and, the hotter the surfaces, the more difficult the deposits are to remove.

In order to overcome the problems of such silicated cleaning compositions, copending US patent application serial number 08/266,175, filed on 27th June 1994 (the contents of which are hereby incorporated by reference) provides an alkaline cleaner composition comprising calcium ions; a surfactant selected from alkylpolyglucosides, amphoteric, polycarboxyls and polyhydroxyls; and an α -hydroxy carboxylic acid such as tartaric acid. Whilst such cleaner compositions are found to be stable, silicate-free and soft metal safe, their utility is limited to compositions free from oxidising agents, such as hypochlorite. It is frequently desirable in the general field of cleaning compositions to use oxidising agents such as sodium hypochlorite, particularly because of their disinfectant properties. A disadvantage with the non-silicated soft metal safe alkaline cleaners described in USSN 08/266,175 is that they are not stable in the presence of such oxidising agents.

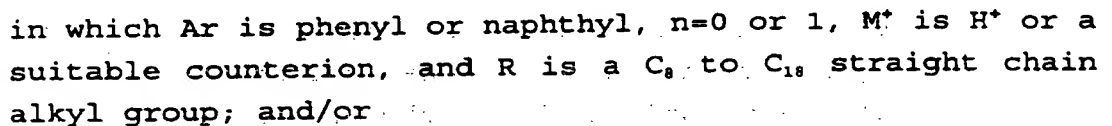
US-A-4229409 describes a method for inhibiting the corrosion of aluminium in contact with aqueous alkaline solutions. 2-phosphonobutane-1,2,4-tricarboxylic acid or the sodium, potassium or ammonium salt thereof is added to the aqueous alkaline solution.

(a) a source of calcium ions;

(b) 2-phosphonobutane-1,2,4-tricarboxylic acid or a salt thereof (PBTC); and

(c) (i) a saturated polymer containing carboxylate groups;

(ii) a sulphonate compound of general formula (I)


$$\begin{array}{c} \text{R}' - (\text{Ar}') - \text{O} - (\text{Ar}'') - \text{R}'' \\ \quad \quad \quad \diagdown \quad \quad \quad \diagup \\ \quad \quad \text{SO}_3^- \text{M}'^+ \quad \quad \text{SO}_3^- \text{M}''^+ \end{array} \quad (\text{II})$$

The concentrate is found to be stable in the presence of both oxidising agents and alkali. This therefore provides a useful alternative to alkaline cleaners containing silicates for use on soft metals such as aluminium.

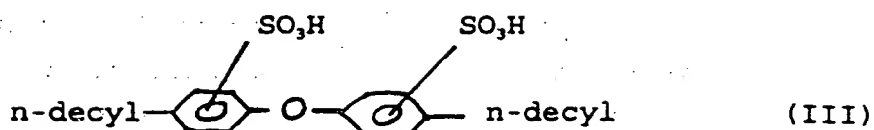
(3)

Component (c) tends to act as a weak sequestrant for the calcium ions. It is therefore important to avoid the presence in the concentrate of any strong chelating agents such as EDTA or its analogues. In an advantageous embodiment, component (c)(i) comprises a polyacrylate polymer. The polymer may be a homopolymer or a copolymer although it has been found that homopolyacrylates are particularly useful. Homopolyacrylates of molecular weight 4,500 are commercially available, such as Norasol LMW45, which is a 50% solution of homopolyacrylate available from Norso Haas. The polyacrylate polymer is preferably present in the concentrate in the range 5% to 7.5% by weight, preferably around 6.5% by weight of the concentrate. Because these saturated polymers tend not to have strong surfactant or foaming properties, it is advantageous to include in the concentrate an additional foaming surfactant. This is discussed in further detail below.

In addition to, or instead of the saturated polymer component (c)(i), the sulphonate compound of general formula (I) or (II) may be incorporated in the concentrate. Preferably, component (c)(ii) comprises the sulphonate compound of general formula (I) in which Ar is phenyl, $n=1$ and R is an n-dodecyl group. This compound is available as a commercial mixture of sulphonates, which sulphonates are substituted at various positions on the phenyl ring. This commercial mixture is sold by Albright and Wilson under the trade name Nansa SSA. Preferably, this sulphonate compound is present in the concentrate in the range 2% to 4% by weight of the concentrate.

Component (c)(iii) preferably comprises the sulphonate compound of general formula (II) in which R' and R'' are both n-decyl groups and Ar' and Ar'' are both phenyl. A sulphonate compound having the general formula (III) as shown below is commercially available from the Dow Chemical Company under the trade name Dowfax 3B2.

(4)



This sulphonate compound is preferably present in the concentrate in the range 3% to 5% by weight of the concentrate.

Each of the sulphonate compounds is not only thought to act to sequester the calcium ions but also provides foaming surfactant properties.

The source of calcium ions can be any conventional source such as a water-soluble calcium salt. Examples of such salts include calcium chloride, calcium nitrate and calcium acetate.

Calcium acetate is a particularly useful source of calcium ions. Preferably, the source of calcium ions provides a calcium ion concentration in the range 0.25 to 0.5% by weight of the concentrate, more preferably around 0.375% by weight of the concentrate.

A suitable source of PBTC is a commercially-available solution of 50% in water available from Bayer under the trade name Bayhibit AM. Preferably, the PBTC is present in the range 0.5% to 3%, more preferably 0.75% to 2.1% by weight of the concentrate, especially around 1.25%.

Whilst the above concentrates can be supplied in the absence of alkali for subsequent mixture with an alkali source, it is preferable that the concentrate further comprises an alkali source, such as an alkali metal hydroxide. Useful alkali metal hydroxides are sodium hydroxide and potassium hydroxide, the potassium hydroxide being preferred. The alkali metal hydroxide is generally present in the range 3 to 5% by weight of the concentrate, preferably around 4.5% by weight of the concentrate.

(5)

A particular advantage of the concentrate according to the present invention is that it can be formulated so as to be stable in the presence of an oxidising agent. The use of oxidising agents in cleaning compositions is advantageous because the oxidising agent can act as a biocide and also as a bleach. Amongst the oxidising agents useable in the present invention are hydrogen peroxide, isocyanurates, persulphates, perborates and halophores which are capable of producing a halogen-containing oxidising species. The halophores include chlorophores, such as hypochlorite, chloramines, chlorine dioxide and perchlorates; bromophores, and iodophores, as well as mixed halides. The chlorophores, such as hypochlorite, are particularly useful as both biocides and bleaches. Thus, advantageously, the concentrate of the present invention further comprises an oxidising agent, preferably a chlorophore. The chlorophore, such as sodium hypochlorite, is typically present in the concentrate in the range 2.2 to 2.8% by weight of the concentrate.

In concentrates containing the oxidising agent, each of the components of the concentrate must be sufficiently stable to the oxidising agent during the typical storage lifetime of the concentrate. Preferably, none of the organic components of the concentrate should contain any regions, such as unsaturated regions, which are capable of being oxidised in the presence of the oxidising agent.

It is advantageous for the concentrate to be able to foam when applied in diluted form to the surface to be treated. As a general rule, the presence of foam in a cleaning operation increases the contact time of the cleaning composition with the surface to be treated and this can increase the effectiveness of the cleaning composition. The foam can also help to remove the soil physically from the surface. It is therefore advantageous for the concentrate to include a foaming surfactant. The sulphonate compounds of general formulae (I) and (II) can both act as foaming surfactants as well as calcium ion sequesterants. Thus, the addition to the sulphonate compounds of further foaming

(6)

surfactants or hydrotropes is optional. It is, however, advantageous to add one or more foaming surfactants to the concentrate when the saturated polymer is present because the saturated polymer is not usually a foaming surfactant. Either or both of the sulphonates components of general formulae (I) and (II) can therefore be included in the concentrate, as well as the saturated polymer. Alternatively, any conventional foaming surfactant can be added to the concentrate, provided that it too is stable in the presence of the oxidising agent. Preferably, the foaming surfactant comprises a C_{12} dimethyl amine oxide although other foaming surfactants such as C_{14} dimethyl amine oxide or lauryl sulphate can be used.

Hydrotropes which may also be added to the concentrate include aryl sulphonates such as sodium toluene sulphonate, sodium xylene sulphonate and phosphate esters e.g. Triton H66 (Union Carbide).

In a further aspect, the present invention provides a cleaning composition comprising a concentrate as described above, diluted with water to form a solution in the range 1% to 10% by volume, preferably around 5% by volume. The water used to dilute the concentrate may be soft water or hard water such as typical town mains tap water, for example at 300 mg/l calcium as calcium carbonate.

The present invention will now be described in further detail, by way of example only, with reference to the following Examples.

In each of the following Examples compositions were tested for their ability to prevent corrosion of aluminium, both in terms of corrosion rate and final appearance of the aluminium, and for their stability. Unless otherwise stated, the amounts of component present in each composition are by weight, the total composition being 100%.

(7)

Corrosion Rat

A test method was used based on the ASTM test method G31. Metal coupons of dimensions 50 x 25 x 0.8 mm of aluminium (SIC or 6082). The dry weight of the metal coupons is determined before and after the test and the test usually carried out in quadruplicate. The coupons are suspended in 500 ml test solution made up in distilled water or a suitable water chemistry such as soft or hard water and the aluminium suspended in the solution for twenty-four hours in a semi-sealed bottle. The weight loss over the twenty-four hour test period is measured thereby to allow the corrosion rate to be calculated in mm/year.

The appearance of the aluminium coupons resulting from the tests is compared visually.

Stability

The compositions are tested for stability on storage at 40°, 4°C and room temperature. Each sample should remain in a single phase with minimal precipitation for at least three months at room temperature or for one month at 4°C or 40°C. A freeze-thaw test is also normally performed and the product should be unaffected for three to six freeze-thaw cycles.

Examples**A) Homopolyacrylat m. wt. 4500 with calcium acetate**

	A1	A2
Water	q.s.	q.s.
Calcium Acetate	0.50	1.00
Homopolyacrylate m. wt. 4500 (50%)	13.00	13.00
Potassium hydroxide (45%)	7.50	7.50
Sodium hypochlorite (11-14%)	20.00	20.00
5% dilution in hard water 300mg/l as CaCO ₃		
pH	9.6	10.5
Corrosion rate mm/yr S1C 24 hrs test	0.07	0.07

Example A simply shows that the presence of homopolyacrylate in a calcium-containing alkaline composition gives a low corrosion rate. The composition was found to be highly stable and did not dull the appearance of the aluminium coupons under test. However, when used in the presence of a surfactant, the composition would be highly unstable and unusable.

(9)

B) Example with PBTC and calcium acetat

	B1	B2	B3
Water	q.s.	q.s.	q.s.
Calcium Acetate	0.80	0.80	0.80
PBTC (50%)	5.00	8.00	10.00
Potassium hydroxide (45%)	10.00	10.00	10.00
Sodium hypochlorite (11-14%)	20.00	20.00	20.00
5% dilution in distilled water			
Corrosion rate mm/yr S1C 24 hrs test	9.54	5.00	1.97

Although the compositions of Example B were relatively stable, high corrosion rates were found except at the highest PBTC concentration.

C) Examples with homopolyacrylate m. wt. 4500 and PBTC

	C1	C2	C3
Water	q.s.	q.s.	q.s.
Calcium Acetate	1.00	1.00	1.00
Homopolyacrylate m. wt. 4500 (50%)	13.00	13.00	13.00
PBTC (50%)	1.50	2.70	4.20
Potassium hydroxide (45%)	10.00	12.50	15.00
Sodium hypochlorite (11-14%)	20.00	20.00	20.00
5% dilution in Hard water 300mg/l as CaCO ₃			
pH	10.9	12.0	12.1
Corrosion rate mm/yr S1C 24 hrs test	7.41	3.08	5.03

(10)

Calcium, homopolyacrylate and PBTC are all present in Example C and give rise to a composition which is relatively stable and which gives some reduction in corrosion rates.

D) Examples with homopolyacrylate, PBTC and n-decyl diphenyl disulphonate (Dowfax 3B2)

	D1	D2	D3
Water	q.s.	q.s.	q.s.
Calcium Acetate	1.00	1.00	1.00
Homopolyacrylate m. wt. 4500 (50%)	13.00	13.00	13.00
PBTC (50%)	1.50	2.70	4.20
n-decyl diphenyl disulphonate (30%)	4.00	4.00	4.00
Potassium hydroxide (45%)	10.00	12.50	15.00
Sodium hypochlorite (11-14%)	20.00	20.00	20.00
5% dilution in Hard water 300mg/l as CaCO ₃			
pH	10.9	12.0	12.3
Corrosion rate mm/yr SlC 24 hrs test	0.28	2.45	5.33

In Example D, both homopolyacrylate and n-decyl diphenyl disulphonate are used and it is found that low corrosion rates can be achieved. The compositions of this Example were relatively stable although the appearance of the coupon on test was dull.

(11)

E) Examples of different levels of calcium acetate in formulation with homopolyacrylate, PBTC and n-decyl diphenyl disulphonate (Dowfax 3B2)

	E1	E2	E3	E4
Water	q.s.	q.s.	q.s.	q.s.
Calcium Acetate	0.50	1.00	1.50	2.00
Homopolyacrylate m. wt. 4500 (50%)	13.00	13.00	13.00	13.00
PBTC (50%)	2.00	2.00	2.00	2.00
n-decyl diphenyl disulphonate (30%)	4.00	4.00	4.00	4.00
Potassium hydroxide (45%)	10.00	10.00	10.00	10.00
Sodium hypochlorite (11-14%)	20.00	20.00	20.00	20.00
5% dilution in Hard water 300mg/l as CaCO ₃				
pH	10.6	10.7	10.8	10.4
Corrosion rate mm/yr S1C 24 hrs test	0.16	2.21	0.08	0.01

The ingredients of Example D were used in this Example E with variation of the calcium concentration. Low corrosion rates were found but the compositions were less stable than those in Example D. The appearance of the coupon was improved, however.

(12)

F) Examples of different levels of alkali in formulation with calcium acetate, homopolyacrylate, PBTC and n-decyl diphenyl disulphonate (Dowfax 3B2)

	F1	F2	F3	F4
Water	q.s.	q.s.	q.s.	q.s.
Calcium Acetate	1.50	1.50	1.50	1.50
Homopolyacrylate m. wt. 4500 (50%)	13.00	13.00	13.00	13.00
PBTC (50%)	2.50	2.50	3.00	3.00
n-decyl diphenyl disulphonate (30%)	4.00	4.00	4.00	4.00
Potassium hydroxide (45%)	10.00	12.00	10.00	10.00
Sodium hypochlorite (11-14%)	20.00	20.00	20.00	20.00
5% dilution in Hard water 300mg/l as CaCO ₃				
pH	10.2	12.0	12.4	12.6
Corrosion rate mm/yr SIC 24 hrs test	0.06	1.64	4.03	4.21

In this Example the quantities of alkali were varied and it was found that the corrosion rate increased concomitantly. The composition was found to become unstable at the higher alkali concentrations.

(13)

G) Examples with different surfactant combinations in formulation with calcium acetate, homopolyacrylate and PBTC

	G1	G2	G3	G4	G5	G6	G7	G8
Water	q.s.	q.s.	q.s.	q.s.	q.s.	q.s.	q.s.	q.s.
Calcium Acetate	1.50	1.50	1.50	1.50	1.50	1.50	1.50	1.50
Homopolyacrylate m. wt. 4500 (50%)	13.00	13.00	13.00	13.00	13.00	13.00	13.00	13.00
PBTC (50%)	2.50	2.50	2.50	2.50	2.50	2.50	2.50	2.50
n-decyl diphenyl disulphonate (30%)		4.00		4.00	4.00	4.00		
Sodium alkane sulphonate (30%)		4.00	4.00		4.00			4.00
C12 dimethyl amine oxide (30%)		4.00	4.00			4.00	4.00	
Potassium hydroxide (45%)	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00
Sodium hypochlorite (11-14%)	20.00	20.00	20.00	20.00	20.00	20.00	20.00	20.00
5% dilution in Hard water 300mg/l as CaCO ₃								
pH	10.0	10.2	10.2	10.0	10.1	10.3	10.4	10.4
Corrosion rate mm/yr S1C 24 hrs test	0.03	0.13	0.02	0.08	0.05	0.09	0.08	0.13

(14)

The corrosion rates were low in Example G although different surfactant combinations were found to give different stability results. The Examples using sodium alkane sulphonate were found to be the least stable whereas those using C₁₂ dimethyl amine oxide were found to be the most stable, with the best coupon appearance.

H) Examples based on the example G4 to optimise the calcium level to maximise the stability

	H1	H2	H3	H4	H5
Water	q.s.	q.s.	q.s.	q.s.	q.s.
Calcium Acetate	1.00	1.20	1.30	1.40	1.50
Homopolyacrylate m. wt. 4500 (50%)	13.00	13.00	13.00	13.00	13.00
PBTC (50%)	2.50	2.50	2.50	2.50	2.50
n-decyl diphenyl disulphonate (30%)	4.00	4.00	4.00	4.00	4.00
C ₁₂ dimethyl amine oxide (30%)	4.00	4.00	4.00	4.00	4.00
Potassium hydroxide (45%)	10.00	10.00	10.00	10.00	10.00
Sodium hypochlorite (11-14%)	20.00	20.00	20.00	20.00	20.00
5% dilution in Hard water 300mg/l as CaCO ₃					
pH	10.6	10.0	10.3	10.3	10.0
Corrosion rate mm/yr S1C 24 hrs test	0.36	0.02	0.06	0.05	0.09

In Example H, the amounts of calcium were varied, based on Example G4. The corrosion rates were found to be very low and the coupon appearance generally acceptable. The stability of the samples was not quite as good as that of Example G4.

(15)

I) Examples based on exampl G7 to optimise the calcium level to maximise the stability

	I1	I2	I3	I4	I5
Water	q.s.	q.s.	q.s.	q.s.	q.s.
Calcium Acetate	1.00	1.20	1.30	1.40	1.50
Homopolyacrylate m. wt. 4500 (50%)	13.00	13.00	13.00	13.00	13.00
PBTC (50%)	2.50	2.50	2.50	2.50	2.50
C12 dimethyl amine oxide (30%)	4.00	4.00	4.00	4.00	4.00
Potassium hydroxide (45%)	10.00	10.00	10.00	10.00	10.00
Sodium hypochlorite (11-14%)	20.00	20.00	20.00	20.00	20.00
5% dilution in Hard water 300mg/l as CaCO ₃					
pH	11.8	10.2	10.4	10.3	10.4
Corrosion rate mm/yr S1C 24 hrs test	1.94	0.06	0.11	0.08	0.08

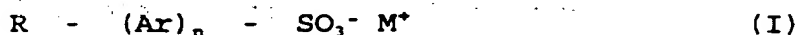
Varying the calcium level based on Example G7, it was found that both stability and coupon appearance were slightly worse than Example G7, with comparable corrosion rates.

(16)

CLAIMS :

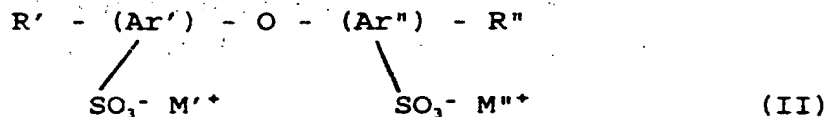
1. A concentrate for use in diluted form in an aqueous cleaning composition, which concentrate comprises the following components:

- (a) a source of calcium ions;
- (b) 2-phosphonobutane-1,2,4-tricarboxylic acid or a salt thereof (PBTC); and
- (c) (i) a saturated polymer containing carboxylate groups;
- (ii) a sulphonate compound of general formula (I)



in which Ar is phenyl or naphthyl, n=0 or 1, M⁺ is H⁺ or a suitable counterion, and R is a C₆ to C₁₈ straight chain alkyl group; and/or

- (iii) a sulphonate compound of general formula (II)



in which Ar' and Ar" are the same or different and are phenyl or naphthyl, M'+ and M"+ are the same or different and are H+ or a suitable counterion, and R' and R" are the same or different and are C₆ to C₁₆ straight chain or branched chain alkyl.

2. A concentrate according to claim 1, wherein component (c) comprises a polyacrylate polymer.

3. A concentrate according to claim 2, wherein the polyacrylate polymer is present in the range 5 to 7.5% by weight of the concentrate.

4. A concentrate according to claim 1, wherein component (c) comprises the sulphonate compound of general formula (I), in which Ar is phenyl, n=1 and R is an n-dodecyl group.

5. A concentrate according to claim 1, wherein component (c) comprises the sulphonate compound of general formula (II), in which R' and R" are both n-decyl groups, and Ar' and Ar" are both phenyl.

6. A concentrate according to claim 4 or claim 5, wherein the sulphonate compound is present in the range 2 to 4% by weight of the concentrate.

7. A concentrate according to any one of the preceding claims, wherein the source of calcium ions provides a calcium ion concentration in the range 0.25 to 0.5% by weight of the concentrate.

8. A concentrate according to any one of the preceding claims, wherein the PBTC is present in the range 0.5 to 3% by weight of the concentrate.

9. A concentrate according to any one of the preceding claims, which further comprises (d) an alkali source.

10. A concentrate according to claim 9, wherein the alkali source comprises an alkali metal hydroxide.

11. A concentrate according to claim 10, wherein the alkali metal hydroxide is present in the range 3 to 5% by weight of the concentrate.

12. A concentrate according to any one of the preceding claims, which further comprises (e) an oxidising agent.

13. A concentrate according to claim 12, wherein the oxidising agent comprises a chlorophore.

(18)

14. A concentrate according to claim 13, wherein the chlorophore comprises a source of hypochlorite.

15. A concentrate according to any one of the preceding claims, which further comprises a foaming surfactant and/or a hydrotrope.

16. A concentrate according to claim 15, wherein the foaming surfactant comprises a C₁₂ dimethyl amine oxide.

17. A cleaning composition comprising a concentrate according to any one of the preceding claims diluted with water to form a solution in the range of 1% to 10% by volume.

18. Use of a cleaning composition of claim 17 in cleaning soft metal surfaces in the presence of an oxidizing agent having biocidal properties.

INTERNATIONAL SEARCH REPORT

Int. Appl. No.
PCT/CA 96/00154

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C23G1/22 C11D3/02 C11D3/36 C11D1/22 C11D1/20
C11D3/37 C11D3/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C23G C11D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	DE,A,19 37 841 (HENKEL & CIE) 12 March 1970 see page 12; claims 1-5; examples ---	1,2,9, 10,12, 17,18
Y	US,A,4 229 409 (SCHARF ROLF ET AL) 21 October 1980 cited in the application see claims 1,4,5; examples ---	1,2,9, 10,12, 17,18
A	US,A,3 653 095 (DUPRE JEAN ET AL) 4 April 1972 see column 1, line 17 - line 24; claims 1,7-14; examples XII, TEST, 10 ---	1,4,9, 10,17
A	EP,A,0 238 728 (NALCO CHEMICAL CO) 30 September 1987 see claims 1,4; examples ---	1
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☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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- * "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- * "&" document member of the same patent family

Date of the actual completion of the international search

25 June 1996

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INTERNATIONAL SEARCH REPORT

International Application No

PCT/CA 96/00154

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/CA 96/00154

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1-3, 7-13, 33, 34

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